

This study investigates the effects of annealing temperature ramp rates on AA7050 aluminum alloy microstructure and hardness for aerospace rivet applications. Samples were annealed at varying ramp rates (2, 4, 6 and 8°/min) and were subjected to mechanical and electropolishing to reveal the microstructure which was analyzed, using Electron Backscatter Diffraction (EBSD). A reduction peripheral coarse grain growth (PCG), or grain and hardness variation in the perimeter of the sample is desired to improve the AA7050's quality.

This work is sponsored by Vista Metals Georgia Adairsville, Georgia

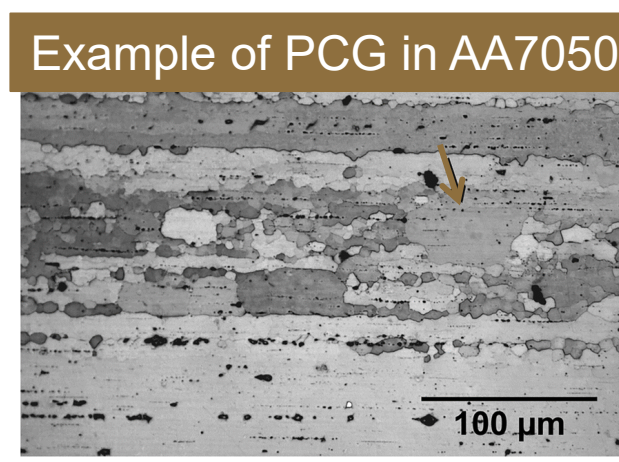


Project Overview

Purpose of Work

Vista Metals supplies AA7050 to customers in the aerospace industry to draw the extruded and hot rolled rod into rivets. The final application of the AA7050 is what determines the rigorous standards this alloy must meet, both mechanically and microstructurally. Rivets must be capable of being compressed to a small size without cracking. As a result, quality is of utmost importance because a variation in the grain structure can become a crack when driving an aluminum rivet. Cracks within the rivets can become a site for failure in the final product.

Peripheral coarse grains (PCGs) are a microstructure defect where the surface layer consists of unusually large recrystallized grains that weaken the mechanical properties and lead to cracks in rivets. PCGs form dynamically during extrusion or statically during annealing. The ability to characterize and manipulate the formation of PCG is crucial to improving AA7050 product quality.



Project Objectives

- Optimize mechanical & electrochemical polishing to visualize the microstructure.
- Determine the effects of annealing temperature ramp rate on the change in hardness during heat treatment, before & after age hardening has occurred.
- Correlate microstructural characteristics to measured hardness across different ramp rates.
- Use grain boundary angle maps & distributions to characterize the amount of recrystallization that has occurred in the product and identify PCG formations.

Methodology

Annealing Procedure

Four 1-inch samples per rod were randomly assigned to different temperature ramp rates (within furnace operating limits), followed by a 4-hour soak at 670°F based on prior Vista Metals optimization. Samples were then air cooled to room temperature.

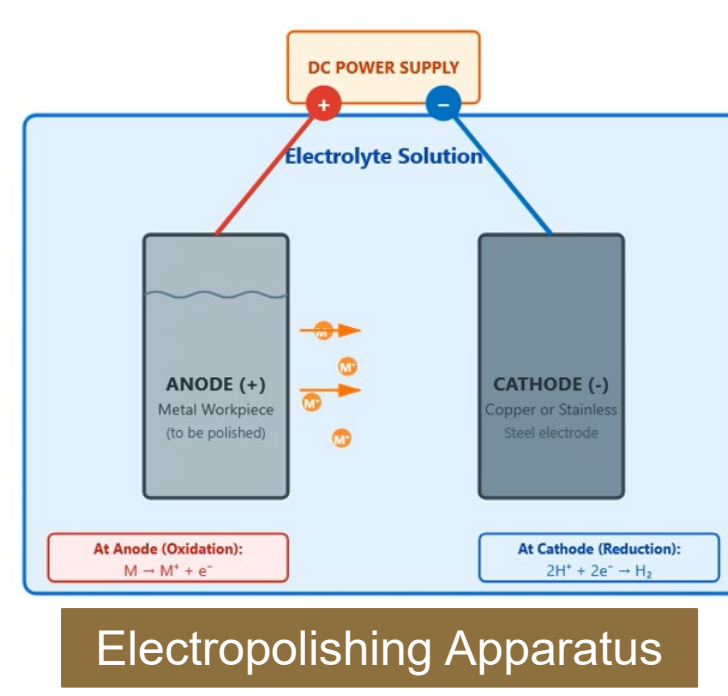
| Annealing Procedure Varying Ramp Rate #2 | | | | |
|--|------------------------------|---------|---------|---------|
| Ramp Rate #1 (Room temperature to 400°F) | 10°F/min | | | |
| Temperature Target #1 | 400°F | | | |
| Ramp Rate #2 (400°F to 670°F) | 2°F/min | 4°F/min | 6°F/min | 8°F/min |
| Temperature Target #2 | 670°F for a 4-hour hold time | | | |

Mechanical Polishing

Samples were polished from 400- 4000 grit SiC then finished with 0.3 μm alumina.

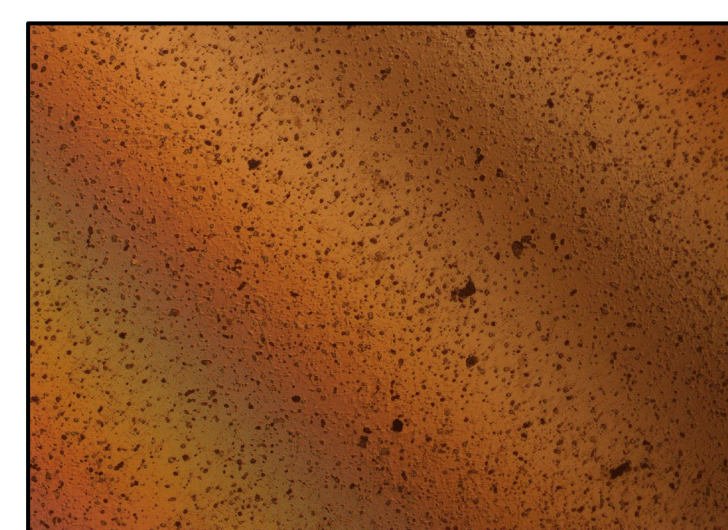
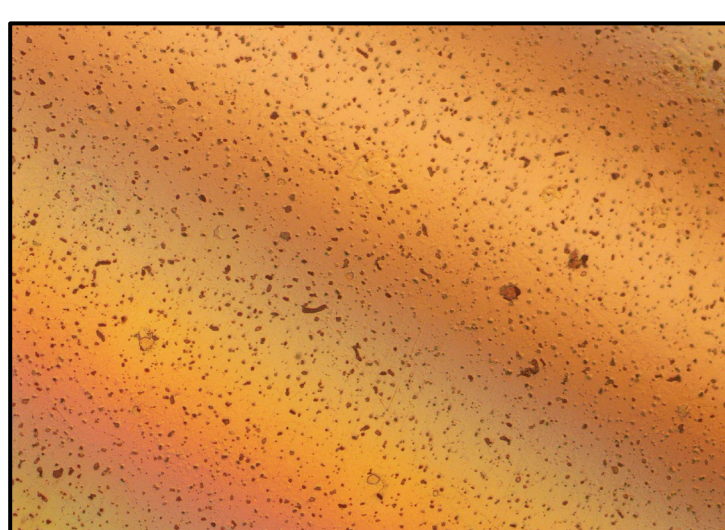
Electropolishing

Electropolishing was utilized to remove the final layer of mechanically induced deformation and anodize the sample surface. Post polishing, samples were rinsed in a sodium bicarbonate solution and DI water to stop residual reactions. Two electrolytes were considered, with Barker's Reagent being preferred due to less pitting under optical microscopy.



Barker's Reagent: A standard solution (ASTM E407) for revealing the grain structure of aluminum alloys under polarized light for grain size evaluation.
 → Experimentally Determined Ideal Polishing Time = 40 sec

Phosphoric Acid: Explored as a safer alternative to perchloric acid based on literature, with an optimal operating voltage found to be 9.5V.
 → Experimentally Determined Ideal Polishing Time = 2.5-5 sec



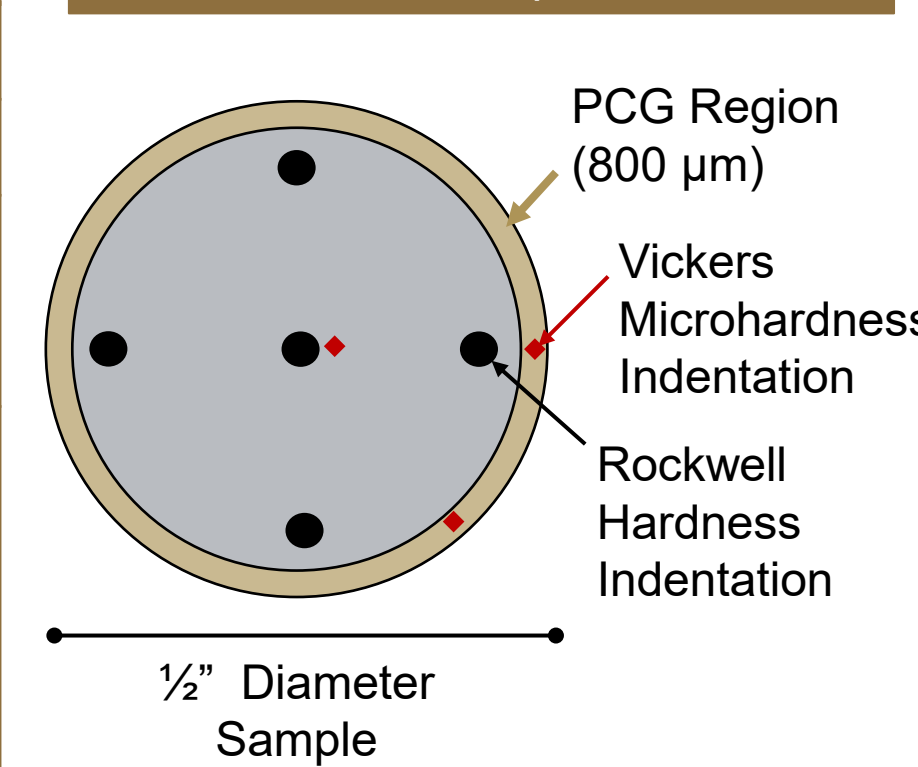
EBSD

Electron backscatter diffraction was performed using an EDAX detector on a Quanta 650 SEM and analyzed with OIM software. Grain boundaries were identified where pixel-to-pixel misorientations exceeded 15°, enabling quantification of recrystallization and grain size distributions. Samples annealed at different ramp rates were characterized in two outer and one center region per sample. Imaging parameters: 20 kV acceleration voltage, spot size 6, working distance ~20 mm.

Hardness Testing

| | Rockwell Hardness | Vickers Hardness |
|-----------------|--|---|
| Scale | HRB | HV0.3 |
| Indenter | 1/16" Ball | 50 μm Diamond |
| Reach PCG Zone? | No | Yes |
| Project Use | Test 5 points on sample surface pre-annealing, post-annealing, and post age hardening to assess change in values | Test center vs perimeter points at locations of EBSD to correlate hardness values to grain size (and presence of PCG) |

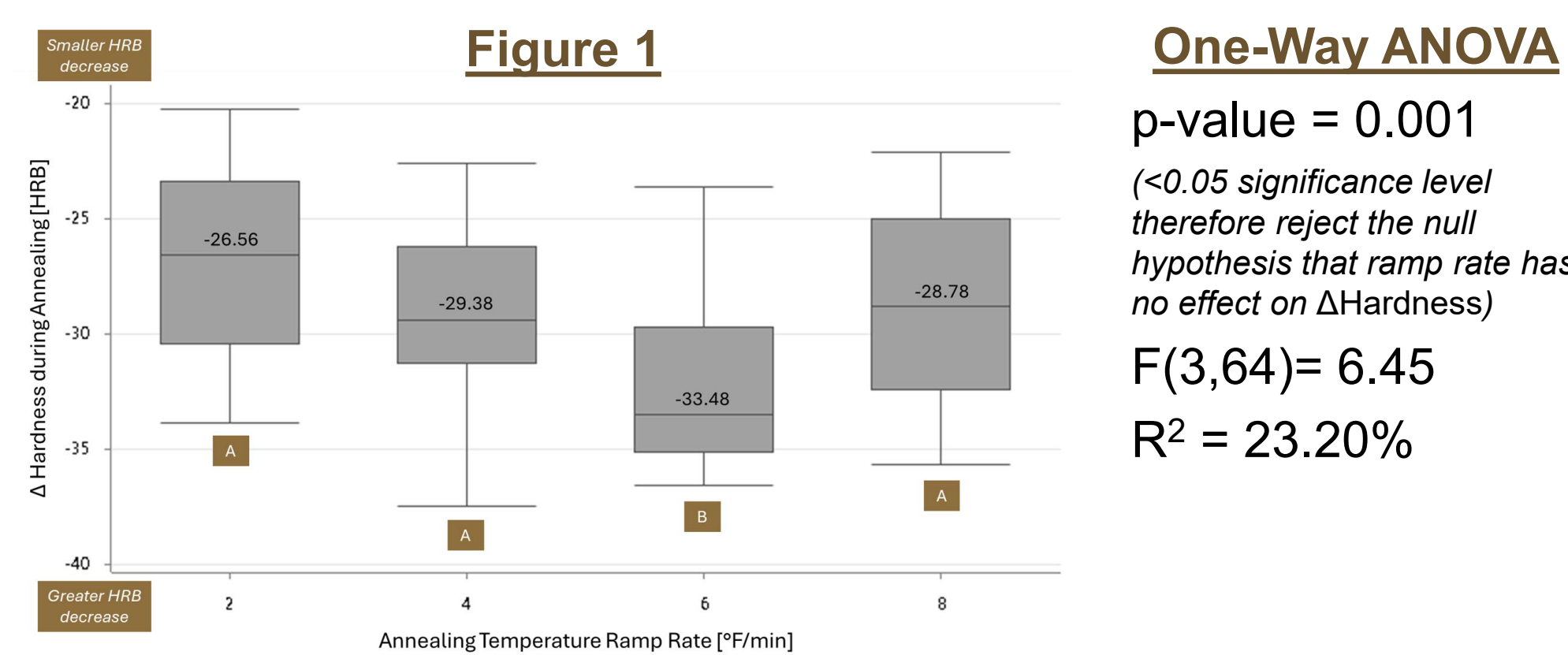
Locations of Hardness Testing vs Region of Peripheral Coarse Grain on Sample



Results & Discussion

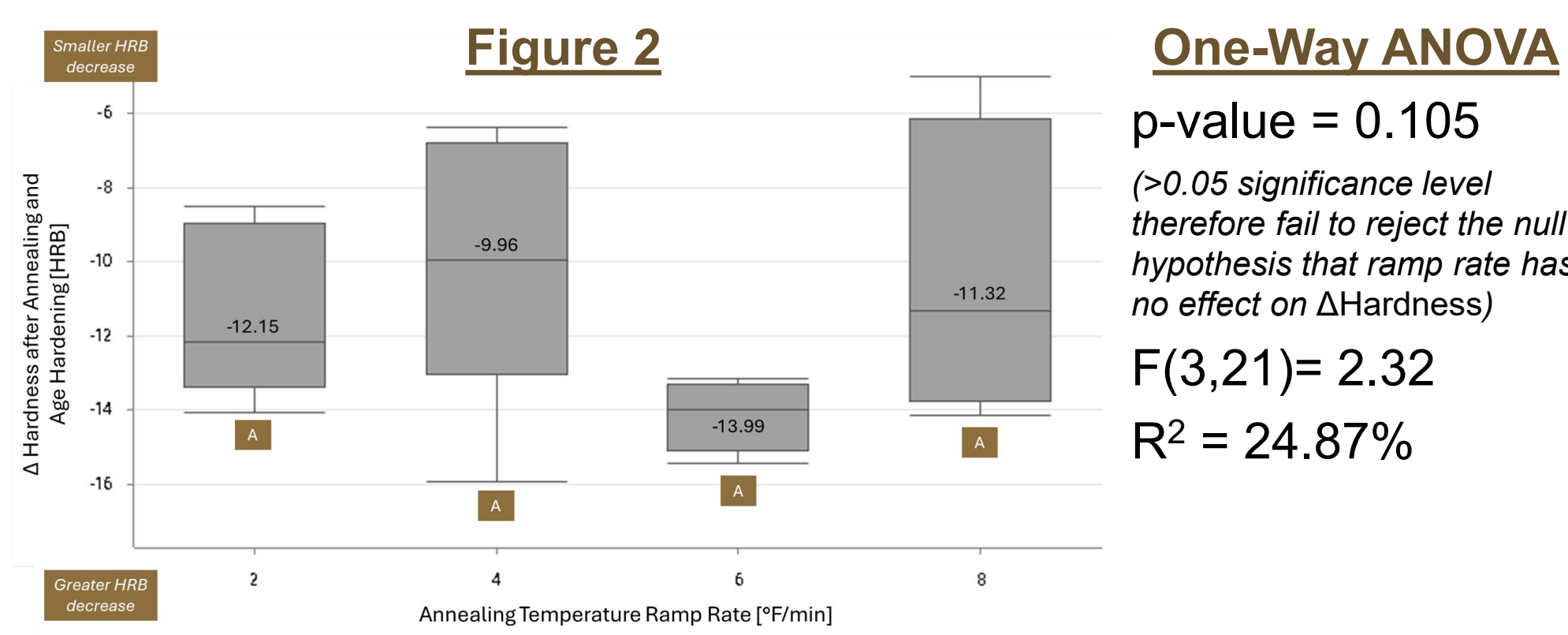
Effect of Ramp Rate on Hardness

Figure 1 shows the change in hardness for 16 AA7050 sections annealed at the four temperature ramp rates, **measured one-day post annealing**. A One-Way ANOVA revealed a significant effect of ramp rate on hardness change (p-value = 0.001). Turkey pairwise comparisons identified **6°F/min as producing a significantly greater hardness reduction than all other ramp rates** (Group B), while 2, 4, and 8°F/min did not differ significantly from one another (Group A).



One-Way ANOVA
 p-value = 0.001
 (<0.05 significance level therefore reject the null hypothesis that ramp rate has no effect on ΔHardness)
 F(3,64) = 6.45
 R² = 23.20%

Figure 2 shows the change in hardness for 6 AA7050 sections per ramp rate **re-tested one-month post-annealing** to assess the effect of age hardening. A One-Way ANOVA found no significant effect of ramp rate on hardness change after age hardening (p-value = 0.105). Turkey pairwise comparisons confirmed **no significant differences between any ramp rate groups** (all four in Group A).



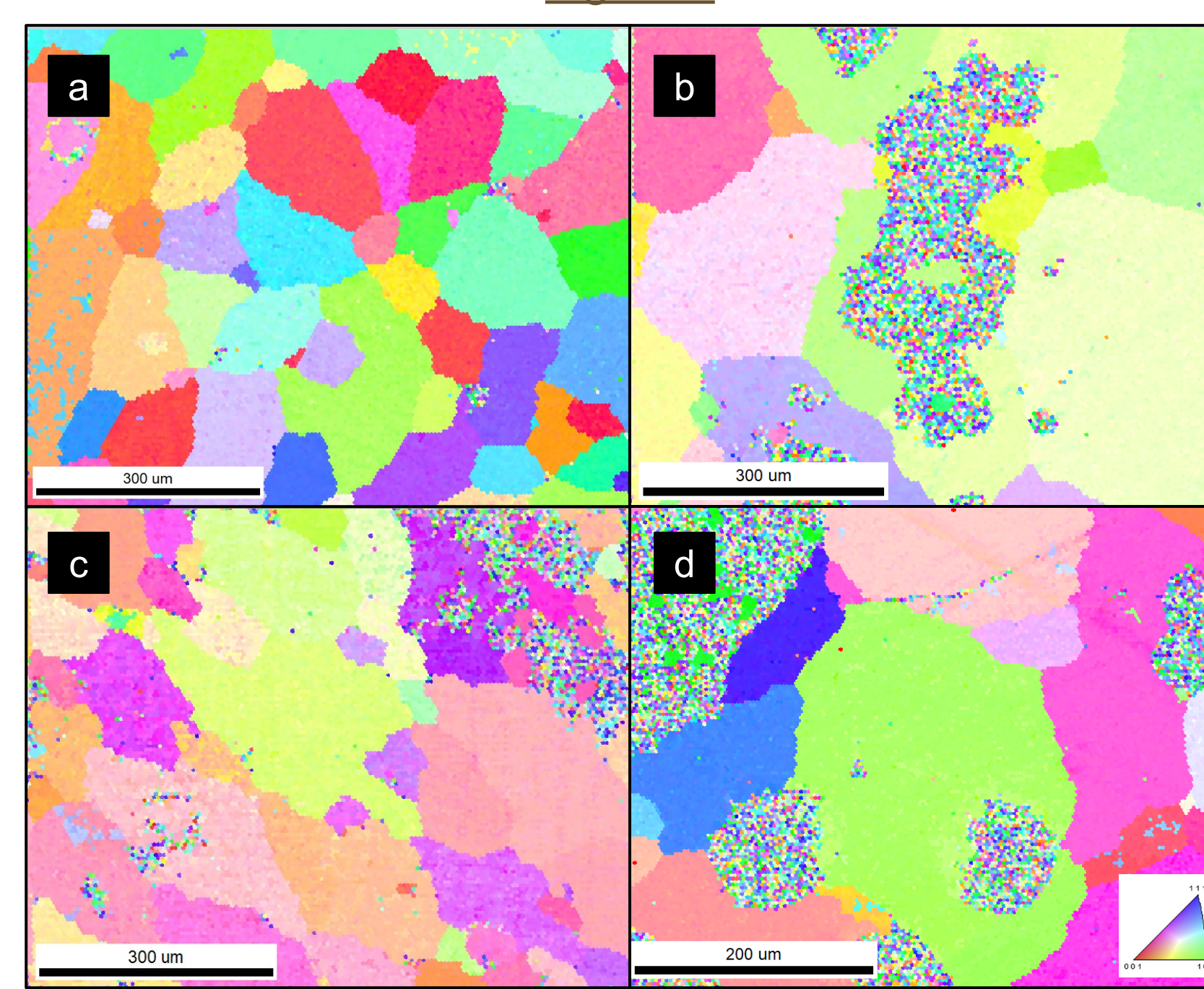
One-Way ANOVA
 p-value = 0.105
 (>0.05 significance level therefore fail to reject the null hypothesis that ramp rate has no effect on ΔHardness)
 F(3,21) = 2.32
 R² = 24.87%

EBSD Inverse Pole Figures

Figure 3 shows inverse pole figures (IPFs) generated from cleaned EBSD data that were taken in the peripheral coarse grain region of the sample. Each grain is colorized according to its crystallographic orientation.

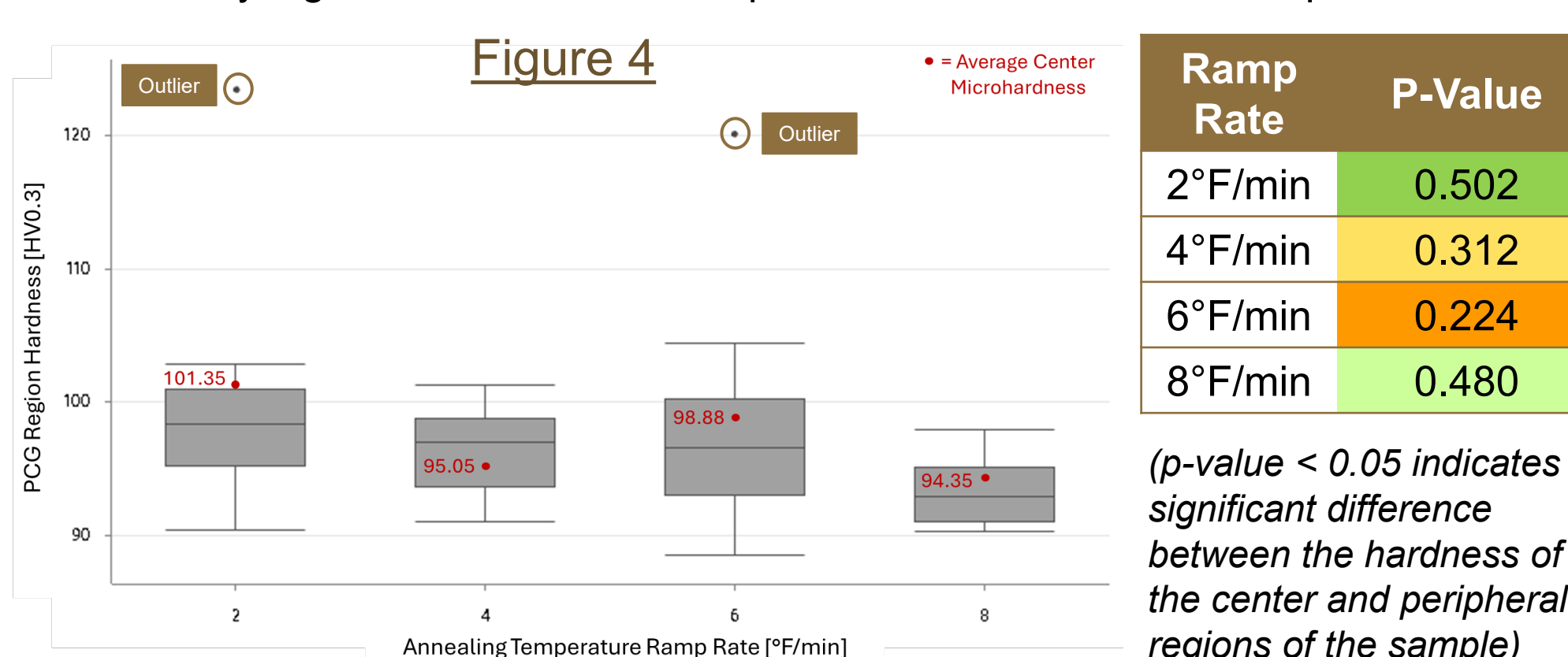
- Images (a) and (b) had the highest CI of the data collected, 0.176 and 0.171, respectively, and were used for subsequent analysis.
- IPF noise is attributed to mechanical deformation and electropolishing artifacts (oxides or pitting), corresponding to regions of low CI.

Figure 3



Hardness in the Peripheral Coarse Grain Region

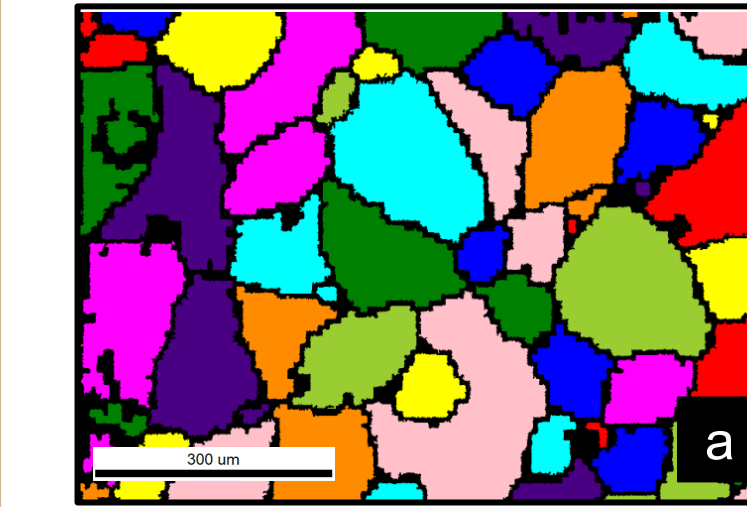
Figure 4 shows a box plot of peripheral microhardness measurements across the four annealing ramp rates, with the average center microhardness overlaid for comparison. Two outliers were identified at 2°F/min and 6°F/min using the 1.5×IQR threshold and excluded from p-value calculations, as their hardness values were substantially higher than the other sample measurements at that ramp rate.



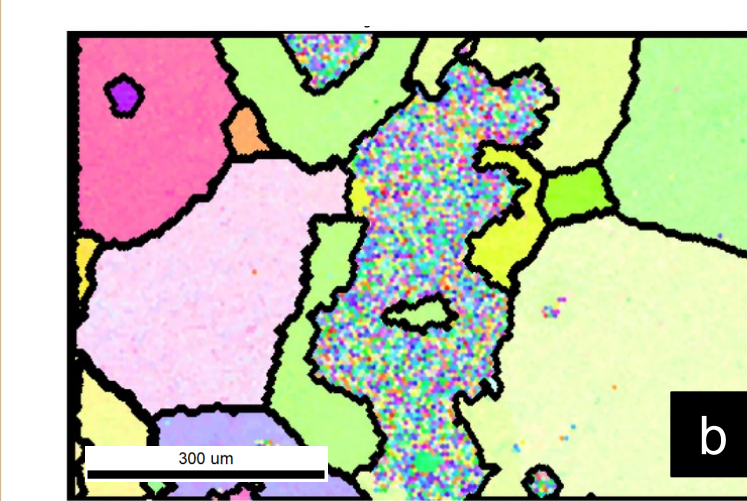
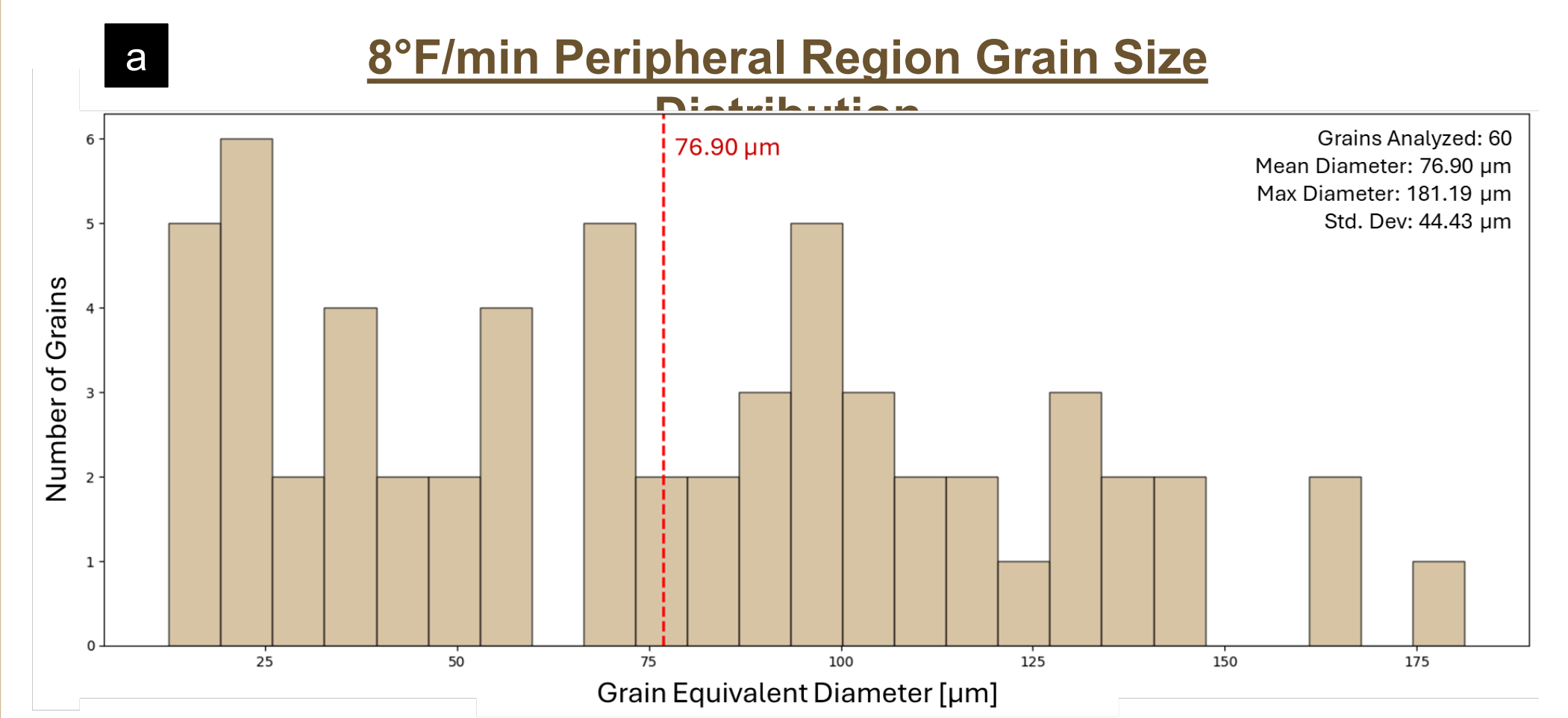
- There was **no statistically significant difference between center and peripheral microhardness** at any ramp rate (all p-values > 0.05).
- The **visual trend of center averages sitting above the perimeter median** (seen in Figure 3) was not statistically confirmed by the t-test.

Results & Discussion

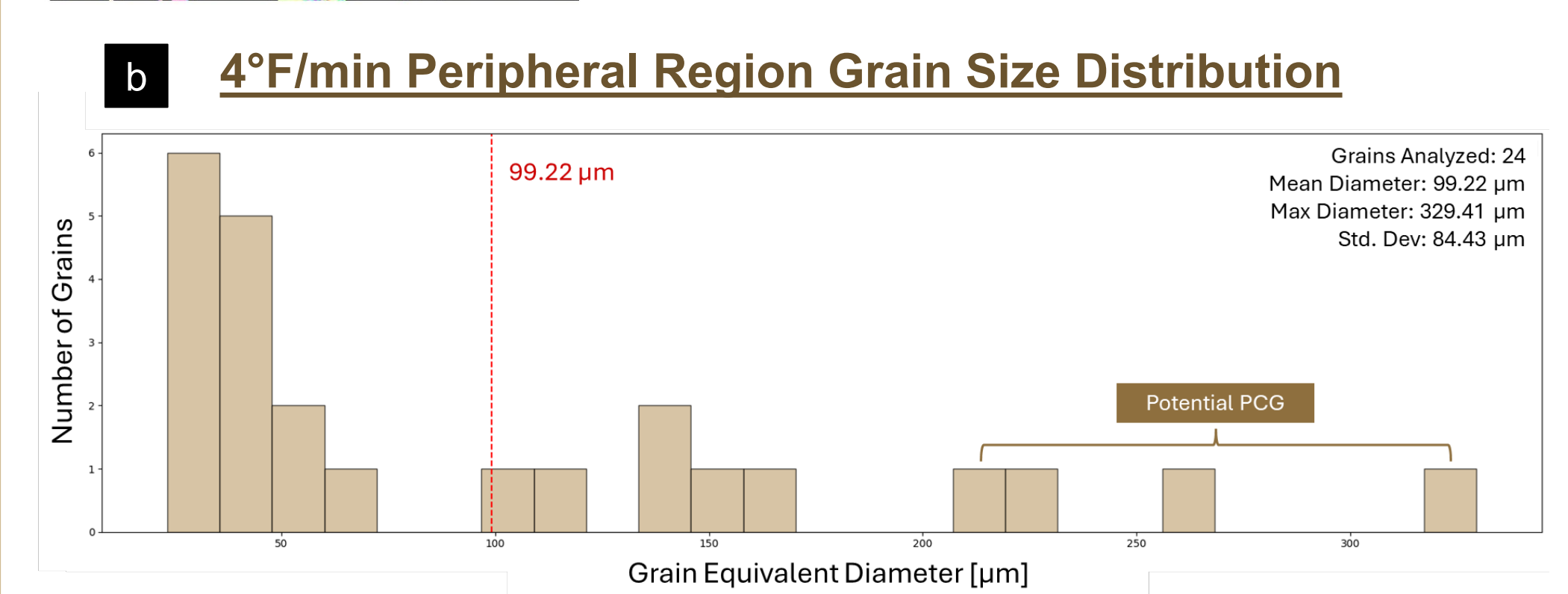
Grain Size Distribution



Grains were segmented using a custom Python script based on their unique color fingerprints. After cleaning the data, equivalent circle diameters were calculated to obtain grain size statistics.



Inverse pole figure of (b) had grains segmented using K means clustering (25) to differentiate grains despite subtle color shifts among them. Equivalent circle diameter was then utilized to obtain grain size statistics.



- Comparing grain size at ramp rates 8°F/min and 4°F/min revealed that the **average grain size increased at a lower ramp rate** (76.90μm vs 99.22μm).
- A lower ramp rate of 4°F/min had larger maximum grain sizes in the perimeter region. **These grains >200μm could be indicative of peripheral coarse grains.**
- Due to noise, sample (b) did not capture as many grains for analysis compared to sample (a), hindering statistical analysis power due to the lower sample size.

Conclusion & Recommendations

- Annealing ramp rate significantly affected hardness one day post-annealing, with 6°F/min producing the greatest softening; however, this effect diminished after age hardening, suggesting that natural aging after a month reduces the influence of ramp rate on final hardness.
- Recommendation:** EBSD could be used at larger fields to image a statistically significant number of grains and regions in each sample to determine ramp rate's effect on microstructure, specifically PCG formation.
- No significant difference was found between center and peripheral microhardness at any ramp rate, indicating PCG did not measurably impact hardness in this study.
 - Recommendation:** Increase the number of hardness measurements taken (within and outside peripheral region) to improve statistical power for detecting PCG effects.
- Lower ramp rates seem to be more prone to produce larger grain sizes (potential PCG) with more variation across sample microstructure as seen in sample (b).
 - Recommendation:** Optimizing polishing to improve sample quality enhances grain detection which may reveal undiscovered trends.
 - Improvements in grain segmentation algorithms would improve statistical quality by removing noise.

Acknowledgements & References

References:

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